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INFORMATION REPORT

REPORT

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COUNTRY East Germany

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SUBJECT Research in and Development of Heavy Water Production at Leuna, Merseburg

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PLACE ACQUIRED

SUPPLEMENT TO REPORT NO.

THIS IS UNEVALUATED INFORMATION

25X1 1. The IG Farben plant at Leuna produced heavy water on a pilot plant scale during World War II. There was no further production of heavy water at Leuna after 1945 because all of the heavy water equipment had been taken to the USSR by that time. The engineer that had installed the heavy water equipment at Leuna, Dr [Carl Ernst] Bode, was among the Leuna heavy water personnel who were taken to the USSR at the end of World War II. [Dr Bode has returned from the USSR and is again employed at Leuna.] 25X1

2. [redacted] the preparation of catalysts for experimental production of heavy water [redacted] was done in a special 25X1 apparatus which consisted of approximately 12 cells, about 1 meter high and 10 cm in diameter, standing upright and arranged in a bank about 1.5 meters in width. Each cell was loosely packed with approximately 1 liter of catalyst. [redacted] Aluminum oxide pellets were dipped in a saturated solution of nickel sulfate and then dried in an oven. This operation was repeated three times and the pellets were then reduced in a stream of 25X1 hydrogen at 450° C to give a catalyst consisting of approximately 30% nickel by weight as an outer coating. The smaller pellets were about 3-7 mm in diameter and the larger ones were about 5-9 mm in diameter.

DOE review completed.

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ARMY	x	AIR	x	FBI		AEC		x	ATIC	x	OSI/WEC & Physics Ev						

25X1 [redacted] This
25X1 heavy water came from Norway. The process was continuous and
25X1 the 4% heavy water flowed into the top of the first cell and after being enriched through the action of the catalyst, flowed out at the bottom and into the top of the second cell and so on until it flowed from the top of the last cell as 98-99% heavy water. The concentration of the heavy water was determined by a hydrometer at the top of the last cell. About 0.01% of gum tragacanth was dissolved in the water and acted as a protective colloid, which prevented granulation of the nickel on the surface of the catalyst. [redacted]

[redacted]

5. In the photocopy showing the two columns (Enclosure (C)), the idea was to cut down on the number of cells by making use of isotope exchange between the hydrogen and water in contact with catalyst at each plate, and also by concentrating the heavy water through fractional distillation at each plate. The condensers at the top of the columns act as a reflux for condensing the steam coming off with the hydrogen. It is to be noted that the hydrogen coming from the top and the water coming from the bottom of column 1 enter the bottom and top respectively of column 2, and vice versa. In the low pressure column at the left, steam at 100° C is probably enriched by the hydrogen, and in the high pressure column at the right, steam at 200° C probably enriches the hydrogen. These columns may be connected in series with other columns operating at higher and lower concentrations of heavy water and the drawing may be for only a single pair of cells in

this series. Obviously, concentration of heavy water could not continue indefinitely by circulating the same water and
25X1 the same hydrogen through the two isolated columns.

25X1 Enclosures (B) and (C) have been included in this report [redacted] because they illustrate the thought and development on this problem going on at Leuna during World War II and were undoubtedly passed on to the Soviets by the Leuna personnel. It should also be noted that there were no titles on the photocopies, and those assigned to the reproductions, Enclosures (B) and (C), are for descriptive purposes only.

ENCLOSURE (A): Translation of Photocopy of Report on Heavy Water Production

ENCLOSURE (B): Diagrams Showing Planned Heavy Water Production Through Isotope-Exchange

ENCLOSURE (C): Diagram Showing Planned Heavy Water Production Through Isotope Exchange in Concentrating Columns

25X1 [redacted]

Main Laboratory
Dept/Experimental Laboratory

Leuna Werke, 2 Oct 45
Dr Geib/Wa.

A DESCRIPTION OF THE DESIGN OF THE CONCENTRATING INSTALLATION FOR HEAVY WATER IN ME 225

(The drawing for the installation is unfortunately not available.)

Water is decomposed by electrolysis. The electrolysis apparatus consists of two cells with polyvinylchloride diaphragms and can hold about two liters of 25% KOH solution. A current of 150 amperes and 10 volts is able to decompose about one liter per hour of water in this apparatus. This yields about 133 liters of hydrogen and 67 liters of oxygen at 20° C and one atm pressure. The two outside plates are water cooled but the inside plate cannot be cooled. The temperature of the KOH solution and the liberated gases is about 40° C. An excess pressure of 0.1 - 0.2 atm develops in the cell due to the liberated gas. This pressure is necessary in order to push the hydrogen through the purifying stages and to overcome the resistance of the catalyst in the reactors to the hydrogen passing through it. Concentrated KOH solution is added to the cells by two igelit tubes entering at the bottom and may be drained in the same manner from the bottom. The tubes are equipped with pinch clamps. The water which is fed into the cells also enters at the bottom and comes from the condenser immediately behind, and connected to, catalyst reactor No 11. The water should enter from as high a point as possible in order to overcome the back pressure and so that the cell can be filled while in operation. The filling line is fitted with a gauge, a valve, and an inspection glass. The hydrogen and oxygen leaving the cells pass through two foam scrubbers to remove the KOH solution which has been mechanically carried along with them. This solution flows back into the chamber of the cell from which it was carried. The pressure difference between the hydrogen and oxygen coming from the electrolytic cells is measured by a difference reading manometer 3. This manometer is filled with water or preferably heavy water or heavy potassium hydrate. In addition, an alarm manometer which rings a bell is installed parallel to manometer 3 to measure the difference in pressure between the hydrogen and oxygen leaving the cell. This is for safety and is auxiliary to manometer 3.

After leaving the foam scrubbers, the hydrogen passes through a dropper into a low temperature cooling vessel which is cooled by means of an inner coil pipe filled with liquid ammonia at atmospheric pressure. The liquid ammonia, taken from the Leuna plant, is introduced into the cooling tube in the required amount by either a float valve or a hand operated valve. The expanded ammonia gas is drawn off from the cooler at atmospheric pressure. This cooling condenses the water vapor in the hydrogen, and ice forms. The exit and entry ports of the cooler are kept open by warming them with steam at about two atmospheres pressure. When the cooler is congested with ice the flow of hydrogen is switched by means of a valve to a second low temperature cooler while the first one is heated with steam to melt the ice, and the water is then drawn off through a sight glass at the bottom.

The hydrogen flows from the low temperature cooler through a separator into a purification chamber that is half filled with platinized asbestos (5% pt.) and heated by a surrounding steam jacket at 16 atmospheres pressure. In the presence of the platinum catalyst, the small amount of oxygen present as an impurity in the hydrogen combines with some of the hydrogen to produce water which can be drawn off through a valve. The separator mentioned above is connected with the outside mercury chamber of the pressure regulator which is used to give the oxygen coming from the electrolysis apparatus the same excess pressure as develops in the hydrogen because of the flow resistance in the evaporators

ENCLOSURE (A): Translation of Photocopy of Report on Heavy Water Production

and catalyst reactors. About 600-900 cc of mercury should be in this pressure regulator and the water which forms in the purification chamber and which collects in the separator should not be allowed to accumulate on the surface of this mercury.

The hydrogen goes from the purifier to the evaporator of stage 11. The evaporator, frequently called a saturator, is heated by means of a steam jacket to a temperature of 90°-100° C and this temperature is measured by an angled thermometer inserted through a connecting line so that the bulb is in the inner space of the evaporator. The temperature can be varied between 50°-120° C by reducing the pressure with a water jet pump or by permitting air to enter through manometer 2, or by increasing the steam pressure to 2-3 atmospheres. At manometer 2 the pressure of the steam which heats all 11 evaporators is controlled by regulating the pressure of the parallel steam heating line. The evaporator is a cylindrical vessel with a volume of about 150 c.c. The hydrogen from the purification vessel goes through a coil wound around the evaporator chamber from top to bottom and enters the bottom of the chamber of evaporator 11. The water from stage 10 enters the top of the chamber of evaporator 11 through a coil around the chamber from bottom to top. Hydrogen and steam in the ratio of 1:1 then reach an equilibrium for the hydrogen isotope exchange in contact with the catalyst of the reactor of stage 11.

Each of the 11 reactors have a space of about 2 liters partly filled with 300 c.c. of catalyst No 6523 in the form of pellets 4 mm in
25X1 diameter. This catalyst contains 30% nickel. [redacted] This may
25X1 be the catalyst described [redacted] in para 2./ The catalyst is placed in a glass frit (Schott) which is fixed in the reactor with a bolted ring. The frit is supported by a sieve plate and the entire assembly is provided with a ring which allows the removal of both the frit and the sieve ring from the reactor. After the catalyst is placed in the reactor, the top is closed with a screw cap provided with an off gas tube, and a built-in frit on the inside of the cap. The reactor is heated at 120°-130° C through a steam jacket containing steam at 2-3 atm pressure. The gaseous mixture coming from the top of the reactor goes downward through a winding condenser tube behind the reactor into a small separator. From this separator the hydrogen, escaping upward, is conducted to the evaporator of the next lower stage; the hydrogen from reactor of stage 11 is conducted to the evaporator of stage 10. On the other hand, the water coming from the separator of one stage drips through a sight glass into the evaporator of the next
25X1 higher stage. In this case, as 11 is the highest stage, the
25X1 water coming from that separator goes to a collecting vessel and
25X1 from there downward through a valve into the electrolytic cells.
25X1 [redacted] Comment: From information [redacted] on the process (paragraph 3), [redacted] it is believed that the water from stage 11 was 95% or better heavy water and the electrolysis was carried out only until the water was decomposed leaving essentially 100% heavy water in the electrolytic cell. The hydrogen from this electrolysis was naturally rich in deuterium and was used to start the cycle through cell 11./

It is necessary that the hydrogen which is obtained by the electrolysis of water coming from stage 11 should run through the system counter-current to the water. Thus the hydrogen runs through the stages in the following sequence: 11, 10, 9, 8, 7, 6, 5, 4, 3, 2, 1. On the other hand, the water flows through the stages in the following sequence: 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, and then to the electrolytic cells. A supply of water containing 10% deuterium is used as a starting material in these

experiments. The water goes from the supply vessel through a measuring tube, a filter, a piston pump, and a settling vessel into the evaporator of stage 1. The collecting vessel, where the water is stored on its way from reactor 11 to the electrolysis cells, serves to prevent the lye from the cells being forced into reactors 11 and 10 by a sudden and unexpected pressure difference between the cells and the reactors during operation. A check valve is unreliable for this purpose.

The oxygen is freed from moisture in exactly the same manner as the hydrogen. It then goes through a pressure regulator supporting a column of mercury, the height of which is determined by the pressure of the hydrogen which is pressing on the large surface of the mercury in the regulator. The oxygen then goes through a large volume buffer where pressures are equalized. The oxygen next goes into a washer filled over half full (level gauge) with water. This is to wash out the KOH vapors that remain in the oxygen. The washer is filled and drained through the bottom and has a valve on top through which the oxygen can be released to the air. The oxygen then goes to a combustion chamber where it is used for burning the hydrogen from stage 1. The hydrogen and oxygen both go through a filter and then through rubber tubes fitted with pinch clamps to a burner which is ignited with a glowing wire. The flame may be observed through a sight glass. The area of the flame is cooled inside and outside with circulating water and the water produced by the flame condenses on these cool surfaces, collects at the bottom and runs through a large U-tube into a collecting vessel. The water produced in this manner contained 5% deuterium, since the hydrogen from which it was made had been in equilibrium with the 10% heavy water used in stage 1. The area of the combustion chamber which is filled before the beginning of the experiment with hydrogen is connected with a water balance (Wasserschaukel) so that the volume of the hydrogen connected with the area of the flame can change considerably. The gas which is not burned by the flame in this operation can pass through the water balance and can enter the line of hydrogen passing by the left side of this device and through the washing bottles into the air. If on the other hand, a surplus of oxygen is created by the loss of hydrogen in the apparatus, hydrogen will change from the left side of the water balance to the right side and will then come to react with oxygen in the flame area. The hydrogen which is constantly bubbling through the washing bottles can be taken from a gas cylinder or from a hydrogen circulating line in the plant.

Every catalyst stage consists of the following:

1. Evaporator heated at 90°-100° C.
2. Catalyst reactor partly filled with 300 c.c. of Leuna Catalyst 3652.
3. Condenser coil with separator and sight glass to observe water dripping into evaporator.
4. Auxiliary pipes and tubing. This includes a valve at the lowest point of the water line leading to the evaporator for drawing off samples for analysis.

The following utilities are necessary to operate this installation:

1. Direct current of 10 volts and 150 amperes for the electrolytic cells.

2. Steam at 2-3 atm pressure to operate the evaporators, reactors, and to heat the gas ports of the low temperature coolers.

3. Steam at 16 atm pressure to heat the hydrogen purification chamber containing platinized asbestos. Water for cooling the condensers, the combustion chamber and the outer electrodes of the electrolytic cells, and water for operating the water jet pump.

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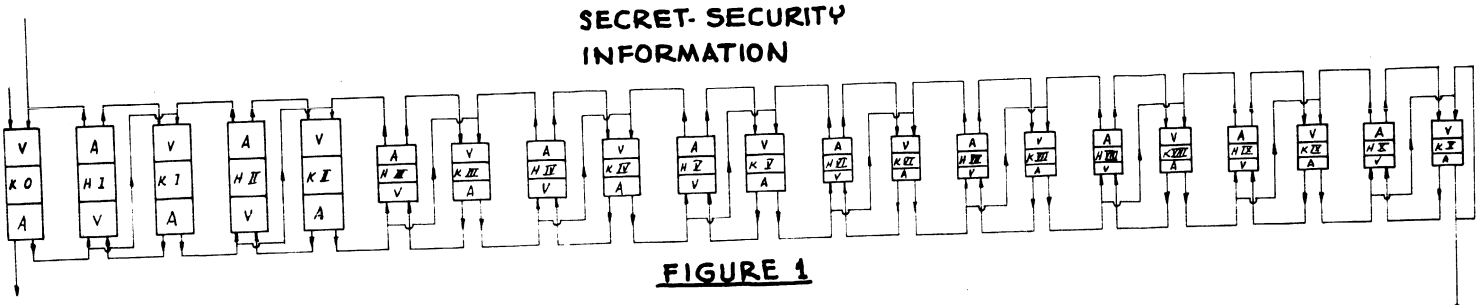

FIGURE 1

Water - Liquid
Water - Vaporized
Hydrogen

V = Vaporization
A = Cooling

K = Cold stage
H = Hot stage

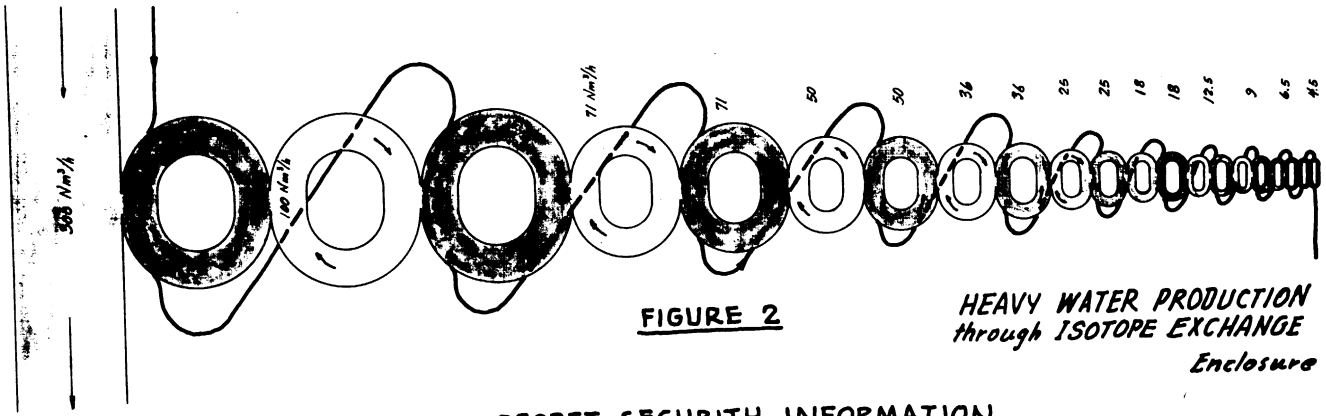

FIGURE 2

HEAVY WATER PRODUCTION
through ISOTOPE EXCHANGE
Enclosure (B)

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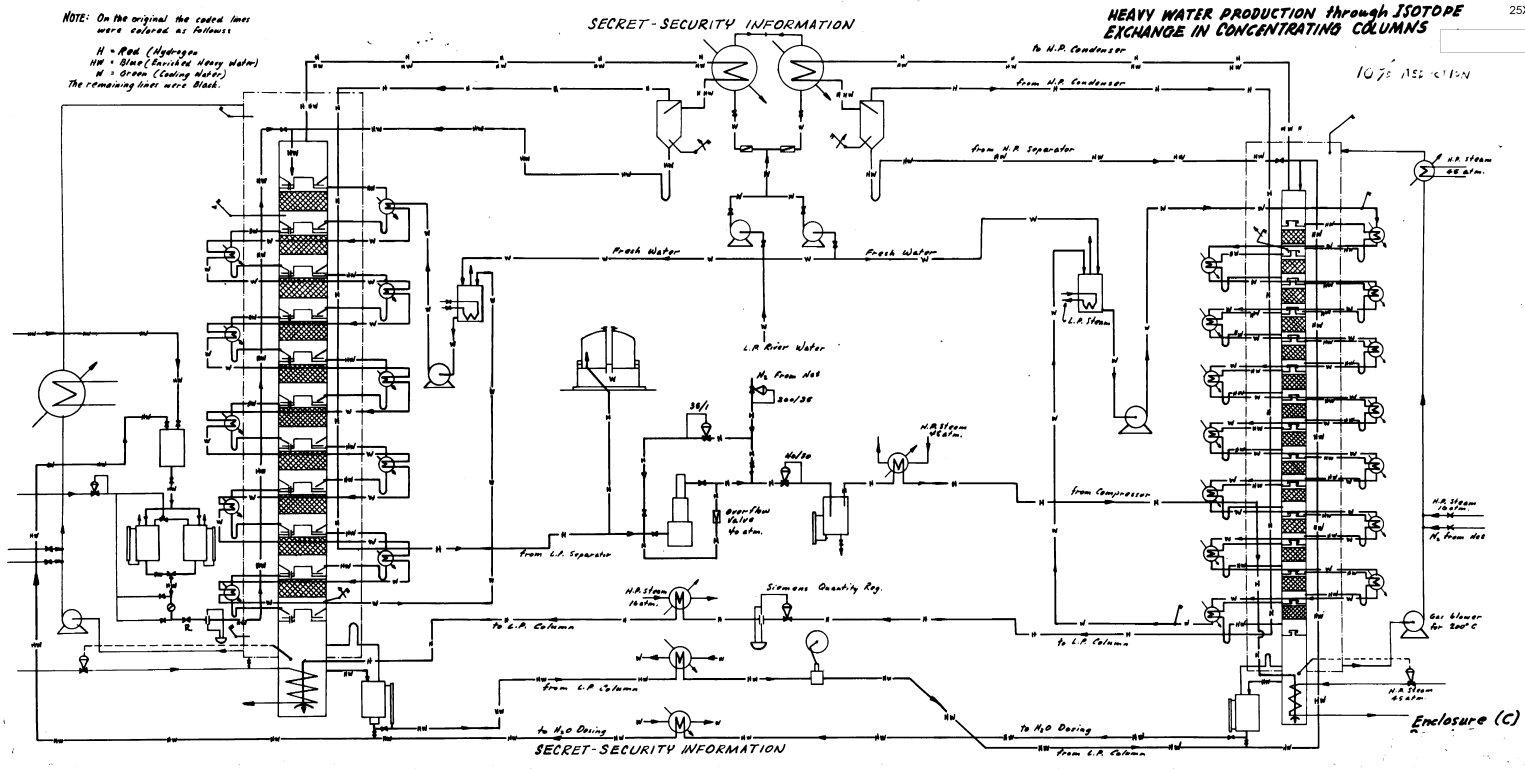
HEAVY WATER PRODUCTION through ISOTOPE EXCHANGE IN CONCENTRATING COLUMNS

NOTE: On the original the coded lines were colored as follows:
H = Red (Hydrogen)
HW = Blue (Enriched Heavy Water)
W = Green (Cooling Water)
The remaining lines were Black.



Enclosure (C)

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